76-10

MAGNETIC BEHAVIOR OF HIGH-DENSITY POWDER METALLURGY BODIES

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER
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ABSTRACT

Magnetic permeability of high-density powder metallurgy bodies was investigated for possible application to nondestructive characterization of residual porosity. The same iron-base bodies were used for measurements of electrical resistivity and ultrasonic velocity.

INTRODUCTION

The current trend in powder metallurgy (PM) is away from the conventional pressed-and-sintered parts with density about 85% of theoretical to parts having densities over 90% or 95%.[1] Densities close to theoretical are essential if PM parts are to be used in critical dynamic components. The dynamic mechanical properties such as toughness and fatigue are highly sensitive to density.[2-4] (Squire's plots[2] have been reproduced in various standard treatments of powder metallurgy including Goetzel, W. D. Jones, and Hirschhorn.) Residual porosity severely impairs these properties, which fall off sharply as the porosity increases. Hence, interest is strong in test methods to qualify PM parts for critical applications by measuring the extent of porosity and establishing that it does not exceed the porosity tolerable for the application.

The physical properties usually providing the basis for nondestructive testing show a relationship with density (or porosity) that approaches linear, especially as the porosity disappears. Among such properties are electrical resistivity and ultrasonic velocity. The static mechanical properties (yield strength, tensile strength, modulus) similarly approach a linear relationship with density. In contrast, the dynamic properties depart strongly from linearity and simulate an exponential relationship. It is obvious that the application of high-density PM parts would be advanced by identification of a physical property having similar nonlinear sensitivity to density and lending itself to evaluation of a part, preferably by a rapid method adaptable to production rates.

A clue to a suitable property was suggested by the doctoral dissertation of Youssef.[5] His work on iron and nickel compacts included magnetic measurements which showed that at low magnetic fields the magnetization increases steeply with density. This sensitivity was related by Youssef to the effect of pores on reversible displacement of domain boundaries.

SCOPE OF PROGRAM

The program was basically concerned with defining the magnetic behavior of a set of ferrous materials, representing a range of densities. The samples were also suitable for simple measurement of mechanical properties such as modulus of rupture and impact resistance. Square bars were prepared from a single type of high-purity iron powder with minimal carbon content, so as to maintain a common microstructure insensitive to any differences in thermal history.

Magnetic measurements were performed with alternating rather than direct current (see Youssef's measurements) to obtain data for a method applicable with

1. *The Trend: Denser, Larger Parts.* Metal Progress, v. 105, no. 4, April 1974, p. 92.
2. SQUIRE, A. *Density as a Criterion of the Mechanical Properties of Iron Powder Compacts.* Watertown Arsenal Laboratory, Experimental Report No. WAL 671/16, October 31, 1944; also, *Density Relationship of Iron-Powder Compacts.* Trans. AIME, v. 171, 1947, p. 485-505.
3. JENKINS, I. *Some Aspects of Residual Porosity in Powder Metallurgy.* Powder Metallurgy, v. 7, no. 13, 1964, p. 68-93.
4. KAUFMAN, S. M., and MOCARSKI, S. *The Effect of Small Amounts of Residual Porosity on the Mechanical Properties of P/M Forgings.* International Journal of Powder Metallurgy, v. 7, no. 3, 1971, p. 19-30.
5. YOUSSEF, H. *Etude des proprietes magnetiques des metaux ferromagnetiques frittes et contribution a l etude de leurs proprietes mecaniques et electriques.* v. 45, 1970, p. 99-121 and p. 140-153; see Chemical Abstracts, v. 73, 1970, 112137p.

reasonable speed in a production line. These measurements required correction for demagnetization factors. The a-c measurements also required consideration of eddy currents as affected by frequency and electrical resistance.

PROCESSING OF SAMPLES

Square bars were prepared with densities ranging from 80% to 100% of theoretical. Available dies guided the selection of sample geometry for compatibility with mechanical (Charpy, transverse rupture) and magnetic measurements. The latter require a length adequate to avoid end effects. Initially, the squares were 0.30 inch on a side with a length of 3.5 inches; samples were prepared with densities of 81, 85, 89, and 93 percent. The die lacked the rigidity to achieve higher density in repressing. Hence, a sturdier die was used for 96% specimens measuring 2.95 x 0.39 x 0.39 inches (as for Charpy tests). For comparison of the dies, additional 86% and 93% specimens were prepared.

Pure carbon-free iron samples were sought to minimize the effects of thermal history and heat treatment atmosphere on microstructure and properties. The starting material was a high-purity commercial atomized iron powder (Smith-Inland grade 300M). Only a minimal amount of lubricant (0.75% Nopco wax) was premixed and this lubricant presumably burned off in the presintering. A preweighed amount of powder for the desired density was compacted in the die and pressed at the tonnage needed for that density. The green pressed bar was preheated and then sintered for 1/2 hour at 2050 F in simulated dissociated ammonia. Densities through 90% were achieved in such a single press-and-sinter cycle. The higher densities (93% and 96%) required recoining followed by resintering.

The same 300M powder was hot forged to obtain stock with essentially full density. The powder was first pressed and sintered, then heated to about 2100 F for forging to a pancake. Square bars of the two sample configurations were machined from this pancake.

Another set of full density specimens, 0.30 inch on a side, was machined from a 7/16-inch-diameter rod of Armco iron. The rod stock had been included in the furnace during the sintering of pressed compacts.

To prevent oxidation, all samples were sprayed with a commercial rust inhibitor (WD-40 or MS-150) as soon as possible after sintering or machining.

The density of each specimen was determined from its measured dimensions and weight. A theoretical density of 7.87 g/cm^3 was assumed. On this basis, samples from the forged pancake had densities of about 7.80 g/cm^3 or 99%; samples from the Armco rod, about 7.83 g/cm^3 or 99.5%.

ELECTROMAGNETIC MEASUREMENTS

Measurements on all samples were made at a frequency of 25 Hz in the test circuit shown schematically in Figure 1. A low frequency was selected in an attempt to minimize the influence of eddy currents on the measurement of magnetic

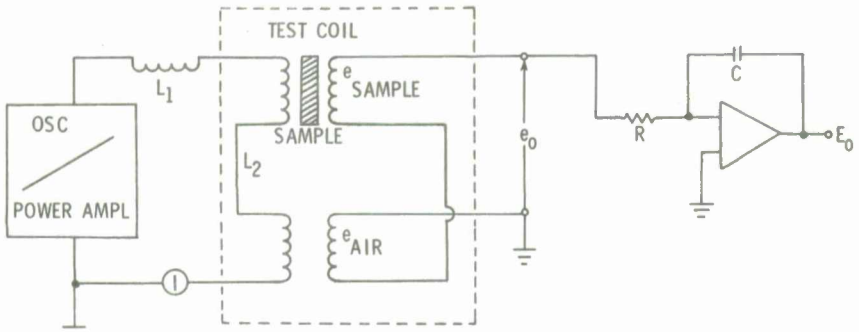


Figure 1. Test circuit for electromagnetic measurements

parameters and was limited at 25 Hz by the availability of instrumentation. In order to approximate a constant-current generator, the inductance L_1 is made very large compared to L_2. The test coil consists of two identical solenoids with primary or field coils connected in series and the secondary windings connected opposing. The primary current establishes an electromagnetic field expressed by

$$H_0 = 4\pi N_1 I K_1 / 10\ell, \tag{1}$$

where N_1 is the primary turns, I is peak current amplitude in amperes, K_1 is a correction factor for short multilayer solenoids, ℓ is the length of the primary winding in centimeters, and H_0 is the resulting field in oersted. When a sample is placed in one of the solenoids, the differential coil arrangement provides an output voltage proportional to the time rate-of-change of flux contributed only by the sample. The induced voltage resulting from primary-to-secondary coupling is eliminated. Inasmuch as the induced secondary voltage e_0 is proportional to the time rate-of-change of flux, the time integral of e_0 performed by the operational amplifier shown in Figure 1 provides an output voltage E_0 proportional to the flux. Therefore, induced magnetism or flux density B resulting from the interaction of the applied field with the sample can be determined by

$$B = RC\sqrt{2} \times 10^8 \, E_0 \, (\text{rms})/N_2 A \tag{2}$$

where RC is the integrator time constant, N_2 is secondary turns, and A is the cross-sectional area of the sample under test.

Because the bar-shaped samples represent an open flux structure, the actual field strength H differs from the applied field H_0 as the result of the demagnetizing field created by the sample itself. This demagnetizing field is nearly proportional to intensity of magnetization and can be characterized by a demagnetizing factor $N/4\pi$ where

$$H = H_0 - (N/4\pi)(B-H). \tag{3}$$

The demagnetizing factor is not only dependent on the sample length-to-diameter ratio but it is also a function of permeability (μ). It was first assumed that

if samples were of equal size in addition to having constant ratio, a constant applied field H_0 would result in an unknown but constant actual field H which would permit comparative measurements. However, initial measurements of μ or B showed that at a constant applied field H_0, the actual field H calculated by Equation 3 varied by almost 50% from lower density to higher density samples, thus demonstrating the influence of permeability on $N/4\pi$.

Therefore, a small flat coil was fabricated and placed at the surface of the test sample within the solenoid for measurement of a voltage proportional to actual field H.

Three voltages are measured: E_0 the time integral of e_0; E_A the time integral of e_{AIR} (Figure 1); and E_H the time integral of the induced voltage of the actual field coil described above. The following magnetic parameters can then be calculated:

$$H_0 \text{ (applied field)} = K_2 E_A \tag{4}$$

$$H \text{ (actual field)} = K_3 E_H \tag{5}$$

$$B \text{ (flux density)} = K_4 E_0/A \tag{6}$$

$$N/4\pi \text{ (demag factor)} = (H_0-H)/(B-H) \tag{7}$$

and $$\mu' \text{ (apparent a-c perm.)} = B/H\,. \tag{8}$$

The K's are constant factors involving integrator time constants and coil geometry. The apparent permeability μ' is an engineering parameter which can be measured accurately at high throughput rates. However, to correct μ' for a-c effects, the frequency of field excitation and electrical properties of the ferromagnetic material must be taken into account. Bozorth[6] shows that the solution to the differential equation governing the penetration of flux into a bar-shaped specimen with an effective diameter d is

$$H = H_0 \left[\frac{\text{ber}^2(2\theta r/d) + \text{bei}^2(2\theta r/d)}{\text{ber}^2\theta + \text{bei}^2\theta}\right] \tag{9}$$

where θ is a dimensionless parameter equal to $\pi d\sqrt{2\mu f}/\rho$, ρ is volume resistivity in abohm-cm, and μ is the true or corrected permeability which shall be referred to as relative permeability μ_{rel}. The ber and bei are the appropriate Bessel functions. It can be shown that when $\theta \geq 4$, $\mu'/\mu = \sqrt{2}/\theta$ and therefore,

$$\mu_{rel} = \pi^2 d^2 \mu'^2 f/\rho. \tag{10}$$

In order to calculate μ_{rel} from Equation 11, the volume resistivity of each sample was first determined. Figure 2 illustrates the standard technique used, based on the equation of ohmic resistance: $R = \rho\ell/A$, where A is cross-sectional area of sample. Since $R = V/I$, $\rho = VA/I\ell$. The potential drop V was measured on all samples at I - 5 amperes from which ρ was then calculated.

6. BOZORTH, R. M. *Ferromagnetism.* D. Van Nostrand Co., N. Y., 1951, p. 775-776.

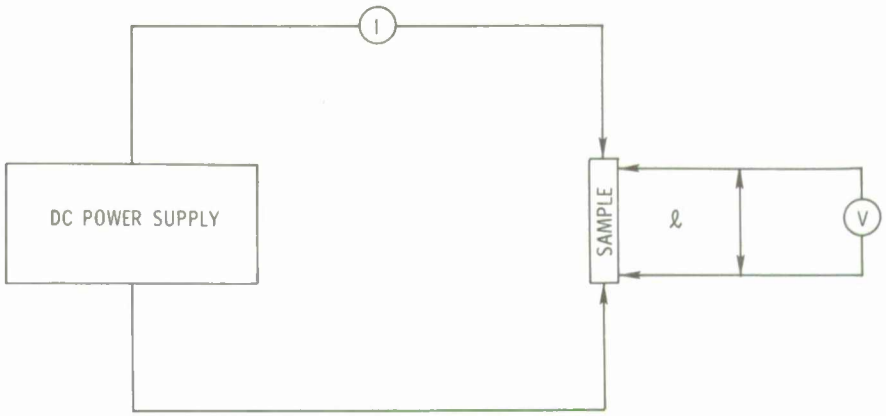


Figure 2. Test circuit for resistivity measurement

Because of significant data scatter in μ' and μ_{rel} versus H and density, the measurement of ultrasonic velocity was undertaken as a referee check on the uniformity of samples not only within groups but also between groups of different dimensions. The ultrasonic test involved the measurement of longitudinal (compressional) wave velocity at 5 MHz on each sample both parallel with and perpendicular to the pressing direction using an accurate pulse-echo superposition technique.[7]

RESULTS AND DISCUSSION

The observed permeability data plotted in Figure 3 are the basis for all the subsequent permeability values in Figures 4 to 7. For convenience the various plots of permeability were separated into pairs representing specimens of the two geometries, i.e., lengths of 3.5 or 2.95 inches. Resistivity data needed for the calculations were determined on the bars and are shown in Figure 8. (The linear relation to density or porosity is confirmed.) Figures 9 and 10 show ultrasonic velocity as a function of density.

The line of reasoning that guided the data processing is brought out through the sequence of permeability plots. Initially an observed or apparent permeability μ' is measured as a function of the actual field H_{act} and the relative density δ, which is the ratio of the actual and theoretical densities. These data are plotted in Figure 3 where the abscissa is H_{act} and δ is the parameter for the families of plots. The μ' is used to calculate the more basic characteristic of each compact, the corrected permeability μ_{rel}, which is plotted in a similar manner in Figure 4. These permeability values are then used in Figures 5 and 6 to derive plots of μ_{rel} versus density at a fixed field at 12.5 Oe or 2.5 Oe. Such plots show the data sought in this program, permeability as a function of density. Each of these figures shows considerable scatter, whose causes are considered below. This scatter casts doubt regarding any generalization about the relation of permeability to density. The apparent relation is more nearly linear than exponential.

7. BROCKELMAN, R. H., *Dynamic Elastic Determination of the Properties of Sintered Powder Metals in Advanced Experimental Techniques in Powder Metallurgy.* Plenum Press, N. Y., 1970.

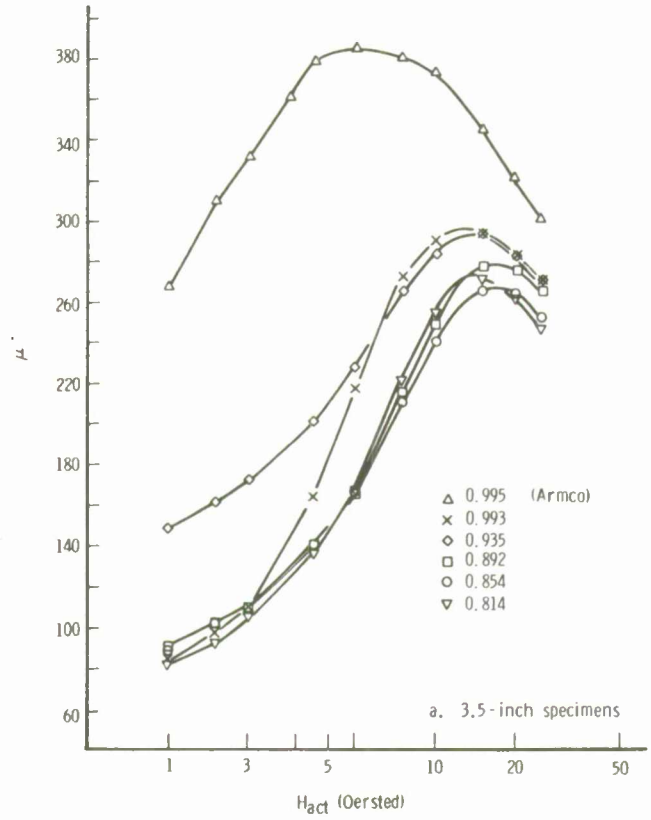
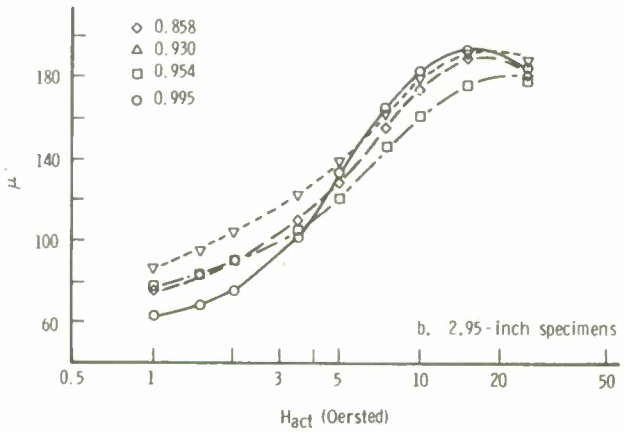


Figure 3. Apparent permeability for various relative densities



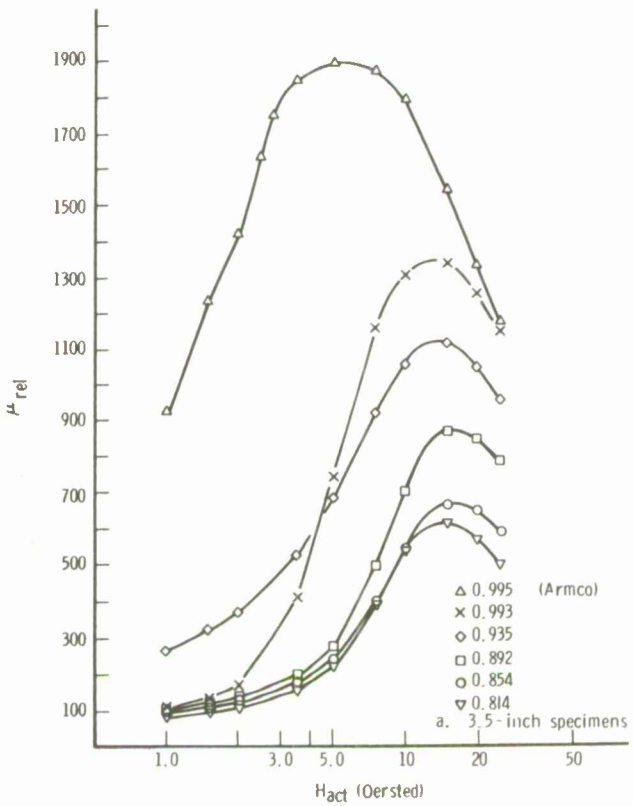
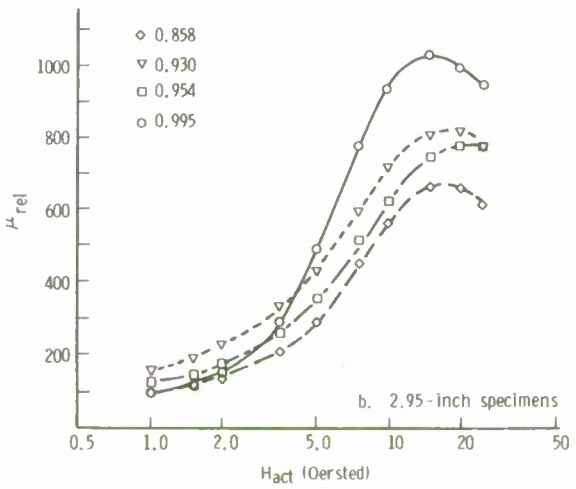


Figure 4. Corrected permeability for various relative densities



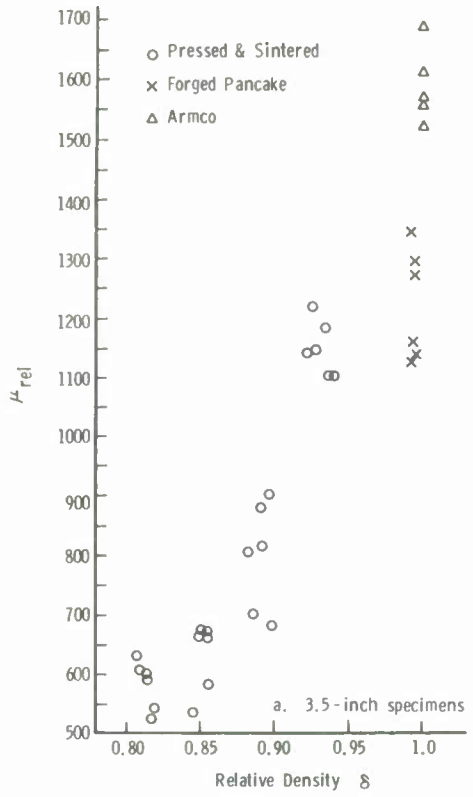
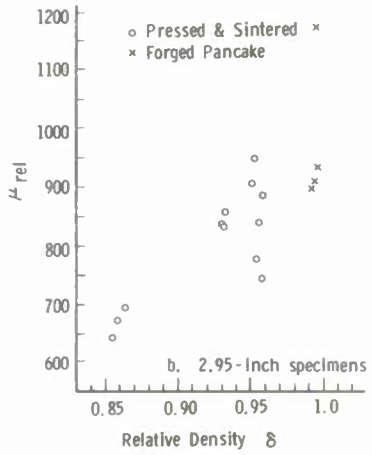


Figure 5. Corrected permeability at 12.5 oersted



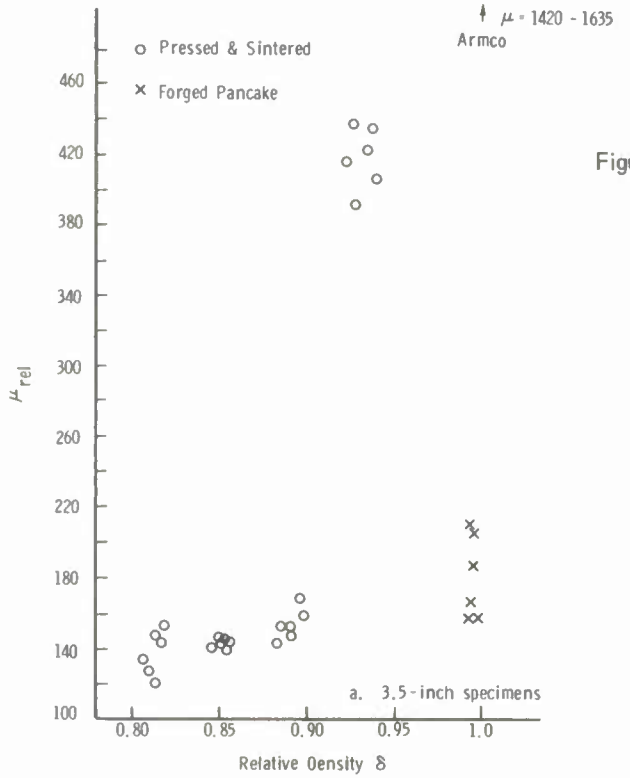
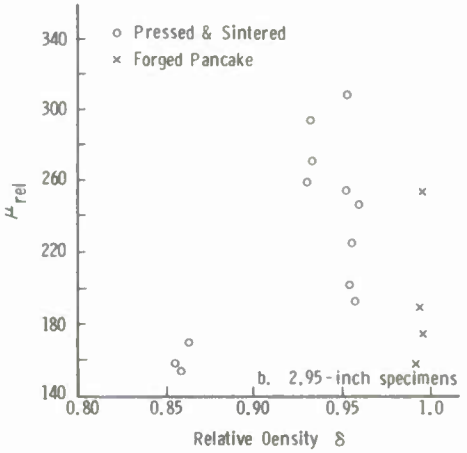


Figure 6. Corrected permeability at 2.5 oersted



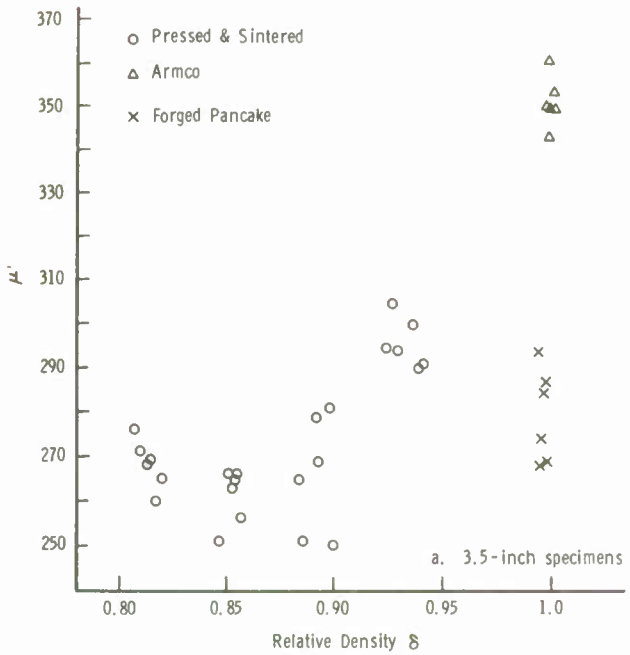
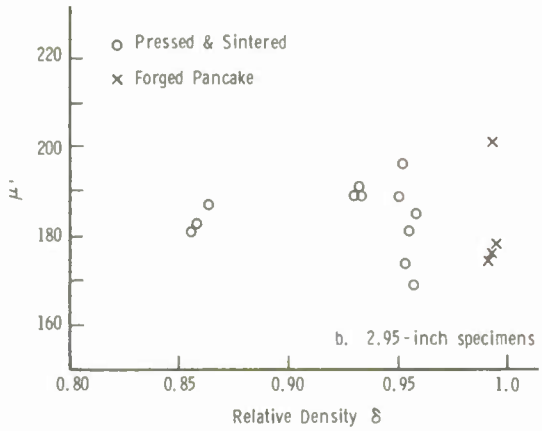


Figure 7. Apparent permeability at 12.5 oersted



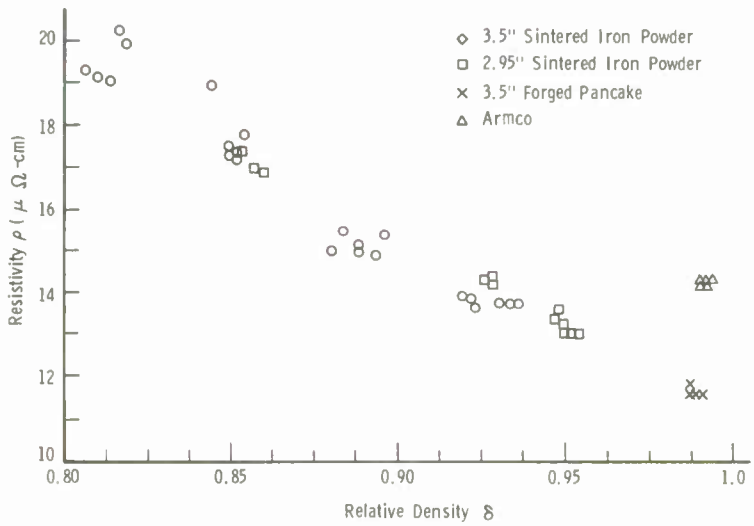


Figure 8. Electrical resistivity of iron

Figure 7 shows similar plots, at 12.5 Oe, of the apparent permeability μ', the quantity that would show up directly in a practical test of a component. Here, too, scatter is the dominant feature. Some glimmer of hope is offered by the sharp increase in μ' from the 90% to the 93% set shown in Figure 7a. But doubt is cast on the high values at 93% by (1) the much lower μ' values for the forged pancake, which has essentially full density, and (2) the μ' values below 200 gauss for the shorter 93% and 95% compacts (Figure 7b). Thus, overall, the magnetic measurements show that magnetic properties are not suitable for evaluation of a ferrous PM component.

The samples were checked by the ultrasonic technique established by Brockelman.[7] His earlier work had shown that ultrasonic velocity correlates not so much with density but rather with tensile strength (see Figure 6 of Ref. 7). Thus, he examined samples of identical density achieved by different routes, so that their mechanical properties differed. The ultrasonic velocity was established as a means of characterizing mechanical behavior as affected by density and other factors, notably pore morphology. The samples used here for the magnetic measurements gave ultrasonic velocities represented in Figures 9 and 10 for directions parallel and perpendicular to the direction of pressing of the compact. The linear correlations demonstrate the value of the ultrasonic method and also inspire confidence in the reliability and consistency of the test samples even without mechanical measurements.

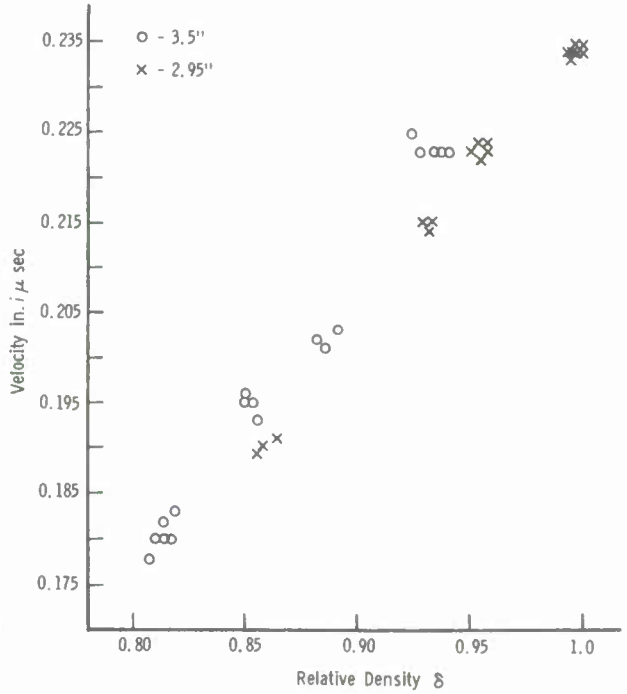


Figure 9. Ultrasonic velocity parallel to pressing direction

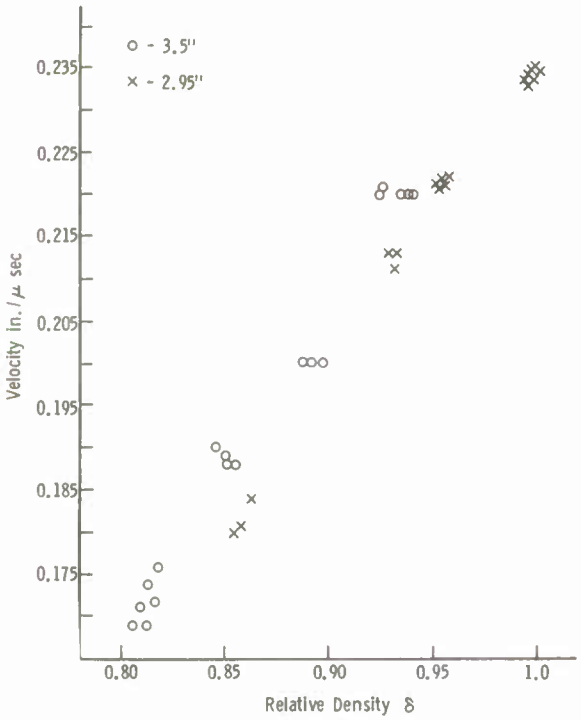


Figure 10. Ultrasonic velocity perpendicular to pressing direction

The factors responsible for the scatter in the permeability data can only be speculated on at this point. Possible differences causing erratic magnetic response could be present in grain size, pore size, pore morphology, and oxide(s), either at the surface or inside a compact.

Several other observations from the measurements are noted here. The Armco iron showed much higher permeability than the forging from powder (Figures 3a, 4a, and 6a). A facile explanation can be presented on the basis of slightly higher density and unquestionably higher purity (deduced from vendor analyses). Yet the Armco material showed high resistivity (15 versus 12 μ ohm-cm - see Figure 8, whereas density and purity should lower resistivity. The Armco material may have its resistivity increased by oxygen, which is presumed to be high. In the ultrasonic measurements (Figures 9 and 10), the two "full-density" materials were indistinguishable.

CONCLUSIONS

Magnetic permeability measurements are not suitable for characterization of residual porosity in high-density bodies prepared from iron powder. Permeability at a fixed field shows linear correlation with porosity but the scatter is considerable. Ultrasonic velocity was confirmed as a more consistent measure of porosity, even though the interrelation is linear rather than a more sensitive relation, such as would be preferred for characterization for critical applications.

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MAGNETIC BEHAVIOR OF HIGH-DENSITY
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Saul Isserow and Harold P. Hatch

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Magnetic permeability of high-density powder metallurgy bodies was investigated for possible application to nondestructive characterization of residual porosity. The same iron-base bodies were used for measurements of electrical resistivity and ultrasonic velocity.

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